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Methyl 2-[(*tert*-butoxycarbonyl)amino]-3-(4-hydroxyphenyl)propanoate

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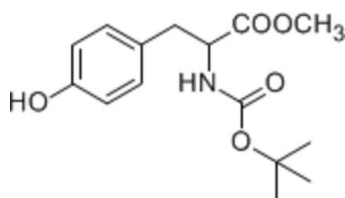
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.039; wR factor = 0.102; data-to-parameter ratio = 19.1.

In the title molecule, $\text{C}_{15}\text{H}_{21}\text{NO}_5$, the dihedral angle between the mean plane of the $-\text{N}-\text{C}(=\text{O})-\text{O}-$ group [maximum deviation = 0.002 (1) Å for the C atom] and the benzene ring is 82.2 (2)°. In the crystal, $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds connect the molecules, forming a two-dimensional network parallel to (001).

Related literature

For the biological activity of related compounds, see: Sykes *et al.* (1999).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{21}\text{NO}_5$

$M_r = 295.33$

Orthorhombic, $P2_12_12_1$

$a = 8.7879$ (8) Å

$b = 9.4844$ (9) Å

$c = 18.9207$ (18) Å

$V = 1577.0$ (3) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.09$ mm⁻¹

$T = 100$ K

$0.55 \times 0.49 \times 0.45$ mm

Data collection

Bruker APEXII CCD diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.962$, $T_{\max} = 0.989$

9339 measured reflections

3636 independent reflections

3469 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.072$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.102$

$S = 1.03$

3636 reflections

190 parameters

H-atom parameters constrained

$\Delta\rho_{\max} = 0.32$ e Å⁻³

$\Delta\rho_{\min} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1B}\cdots\text{O3}^i$	0.86	2.27	3.0583 (16)	153
$\text{O1}-\text{H1C}\cdots\text{O5}^{ii}$	0.82	1.92	2.7356 (15)	180

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, -z$; (ii) $x, y + 1, z$.

Data collection: APEX2 (Bruker, 2007); cell refinement: APEX2; data reduction: SAINT (Bruker, 2007); program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL and PLATON (Spek, 2009); software used to prepare material for publication: SHELXTL and publCIF (Westrip, 2010).

The author thanks Henan University of Traditional Chinese Medicine for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5632).

References

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supplementary materials

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Methyl 2-[(*tert*-butoxycarbonyl)amino]-3-(4-hydroxyphenyl)propanoate

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Comment

Amide, ester and hydroxyl groups widely exist in many biologically active compounds or can be utilized in prodrugs (Sykes *et al.*, 1999). Herein we report the crystal structure of the title compound. The dihedral angle between the mean-plane of the amide group (N1/C9/O5/O4) [a maximum deviation of 0.002 (1)° for C9] and the benzene ring (C1–C6) is 82.2 (2)°. In the crystal, O—H···O and N—H···O hydrogen bonds connect molecules forming a two-dimensional network parallel to (001) (Fig. 2).

Experimental

The title compound (0.3 mmol, 88.5 mg) was dissolved in 10 ml of methanol solution. Colorless block-shaped crystals separated after 5 d.

Refinement

H atoms were included in calculated positions and treated as riding atoms: C—H = 0.93, 0.96 and 0.98 Å for CH(aromatic), CH₃ and CH(methine) H atoms, respectively, or N—H = 0.86 Å and O—H = 0.82 Å with $U_{\text{iso}}(\text{H}) = kU_{\text{eq}}(\text{parent C-atom, N})$, where $k = 1.5$ for CH₃ and hydroxyl H atoms and $k = 1.2$ for all other H atoms. The absolute configuration could not be determined from the X-ray data. In the absence of anomalous dispersion effects Friedel pairs were merged.

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *pubCIF* (Westrip, 2010).

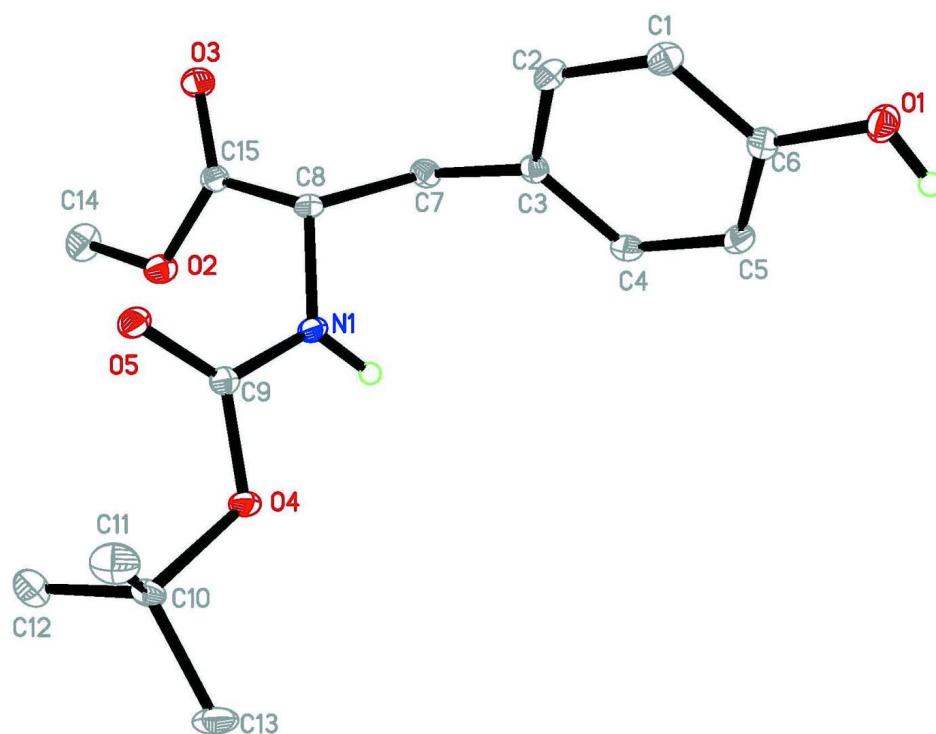


Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

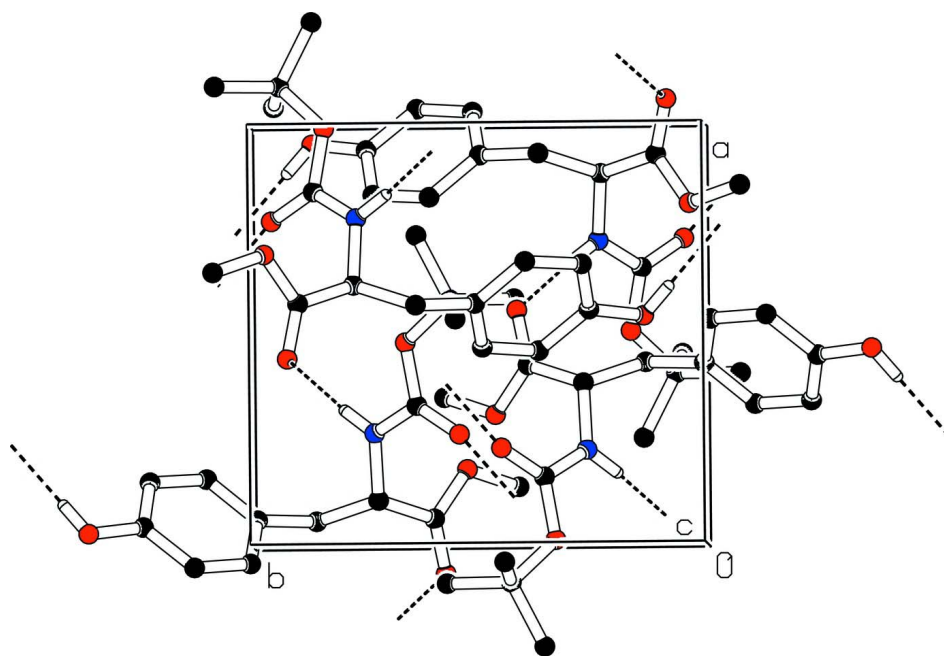


Figure 2

Part of the crystal structure with hydrogen bonds shown as dashed lines. H atoms bonded to C atoms are not shown.

Methyl 2-[(*tert*-butoxycarbonyl)amino]-3-(4-hydroxyphenyl)propanoate

Crystal data

$C_{15}H_{21}NO_5$	$F(000) = 632$
$M_r = 295.33$	$D_x = 1.244 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: P 2ac 2ab	Cell parameters from 9371 reflections
$a = 8.7879 (8) \text{ \AA}$	$\theta = 1.0\text{--}27.6^\circ$
$b = 9.4844 (9) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 18.9207 (18) \text{ \AA}$	$T = 100 \text{ K}$
$V = 1577.0 (3) \text{ \AA}^3$	Block, colourless
$Z = 4$	$0.55 \times 0.49 \times 0.45 \text{ mm}$

Data collection

Bruker APEXII CCD	9339 measured reflections
diffractometer	3636 independent reflections
Radiation source: fine-focus sealed tube	3469 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.072$
φ and ω scans	$\theta_{\text{max}} = 27.6^\circ$, $\theta_{\text{min}} = 2.2^\circ$
Absorption correction: multi-scan	$h = -11 \rightarrow 9$
(<i>SADABS</i> ; Sheldrick, 1996)	$k = -12 \rightarrow 8$
$T_{\text{min}} = 0.962$, $T_{\text{max}} = 0.989$	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H-atom parameters constrained
$wR(F^2) = 0.102$	$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.1712P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
3636 reflections	$(\Delta/\sigma)_{\text{max}} = 0.008$
190 parameters	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	−0.04196 (17)	1.13511 (15)	0.18071 (7)	0.0216 (3)
H1A	−0.1123	1.1434	0.2172	0.026*
C2	−0.03335 (16)	1.01206 (15)	0.14197 (7)	0.0205 (3)
H2A	−0.0978	0.9376	0.1531	0.025*
C3	0.07017 (15)	0.99675 (14)	0.08627 (7)	0.0172 (3)

C4	0.16908 (15)	1.10800 (15)	0.07254 (7)	0.0192 (3)
H4A	0.2409	1.0991	0.0367	0.023*
C5	0.16250 (16)	1.23229 (15)	0.11142 (7)	0.0200 (3)
H5A	0.2297	1.3055	0.1016	0.024*
C6	0.05528 (16)	1.24689 (14)	0.16488 (7)	0.0184 (3)
C7	0.06700 (17)	0.86702 (15)	0.04022 (7)	0.0188 (3)
H7A	0.1336	0.8823	0.0001	0.023*
H7B	−0.0354	0.8547	0.0221	0.023*
C8	0.11575 (14)	0.73007 (14)	0.07772 (7)	0.0166 (3)
H8A	0.0573	0.7217	0.1216	0.020*
C9	0.33960 (15)	0.64306 (14)	0.13963 (7)	0.0159 (3)
C10	0.59030 (15)	0.56854 (15)	0.18449 (7)	0.0199 (3)
C11	0.5429 (2)	0.5555 (2)	0.26171 (8)	0.0302 (3)
H11A	0.5434	0.6471	0.2833	0.045*
H11B	0.6131	0.4950	0.2861	0.045*
H11C	0.4424	0.5161	0.2644	0.045*
C12	0.59178 (18)	0.42703 (17)	0.14678 (8)	0.0280 (3)
H12A	0.6225	0.4403	0.0985	0.042*
H12B	0.4917	0.3866	0.1480	0.042*
H12C	0.6621	0.3649	0.1699	0.042*
C13	0.74355 (17)	0.64103 (19)	0.17817 (9)	0.0305 (3)
H13A	0.7714	0.6480	0.1292	0.046*
H13B	0.8190	0.5872	0.2030	0.046*
H13C	0.7373	0.7338	0.1982	0.046*
C14	0.1469 (2)	0.42237 (17)	−0.04498 (8)	0.0292 (3)
H14A	0.2370	0.3749	−0.0609	0.044*
H14B	0.0943	0.4618	−0.0848	0.044*
H14C	0.0816	0.3563	−0.0213	0.044*
C15	0.07176 (16)	0.60783 (14)	0.02939 (7)	0.0180 (3)
N1	0.27499 (13)	0.73700 (12)	0.09567 (6)	0.0170 (2)
H1B	0.3301	0.8029	0.0778	0.020*
O1	0.04029 (13)	1.36708 (11)	0.20398 (5)	0.0247 (2)
H1C	0.1090	1.4220	0.1939	0.037*
O2	0.18880 (11)	0.53485 (11)	0.00379 (5)	0.0221 (2)
O3	−0.05943 (13)	0.58394 (11)	0.01458 (6)	0.0254 (2)
O4	0.48955 (11)	0.66726 (10)	0.14566 (5)	0.0186 (2)
O5	0.27010 (12)	0.54940 (10)	0.16994 (5)	0.0204 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0215 (7)	0.0263 (7)	0.0171 (6)	−0.0023 (6)	0.0044 (5)	0.0007 (5)
C2	0.0205 (6)	0.0205 (6)	0.0206 (6)	−0.0054 (5)	0.0015 (5)	0.0015 (5)
C3	0.0161 (6)	0.0182 (6)	0.0172 (6)	0.0019 (5)	−0.0039 (5)	0.0011 (5)
C4	0.0145 (6)	0.0235 (7)	0.0195 (6)	0.0009 (5)	0.0020 (5)	0.0009 (5)
C5	0.0176 (6)	0.0208 (6)	0.0217 (6)	−0.0034 (5)	0.0006 (5)	0.0018 (5)
C6	0.0200 (6)	0.0185 (6)	0.0167 (5)	−0.0002 (5)	−0.0023 (5)	0.0006 (5)
C7	0.0179 (6)	0.0208 (6)	0.0178 (6)	0.0018 (5)	−0.0025 (5)	−0.0009 (5)
C8	0.0116 (6)	0.0203 (6)	0.0178 (6)	−0.0004 (5)	−0.0014 (5)	−0.0005 (5)
C9	0.0152 (6)	0.0171 (6)	0.0155 (5)	−0.0002 (5)	0.0007 (5)	−0.0038 (5)

C10	0.0145 (6)	0.0248 (7)	0.0206 (6)	0.0027 (5)	−0.0050 (5)	0.0017 (5)
C11	0.0281 (8)	0.0429 (9)	0.0196 (6)	0.0005 (7)	−0.0040 (6)	0.0012 (6)
C12	0.0281 (8)	0.0264 (7)	0.0296 (7)	0.0064 (6)	−0.0033 (6)	0.0000 (6)
C13	0.0146 (7)	0.0421 (9)	0.0348 (8)	−0.0032 (6)	−0.0069 (6)	0.0052 (7)
C14	0.0364 (9)	0.0281 (7)	0.0232 (7)	−0.0031 (7)	0.0026 (6)	−0.0084 (6)
C15	0.0164 (6)	0.0189 (6)	0.0188 (6)	−0.0003 (5)	−0.0011 (5)	0.0037 (5)
N1	0.0129 (5)	0.0185 (5)	0.0196 (5)	−0.0023 (4)	−0.0008 (4)	0.0012 (4)
O1	0.0269 (5)	0.0215 (5)	0.0256 (5)	−0.0057 (4)	0.0063 (4)	−0.0059 (4)
O2	0.0191 (5)	0.0249 (5)	0.0224 (5)	−0.0012 (4)	0.0020 (4)	−0.0060 (4)
O3	0.0179 (5)	0.0250 (5)	0.0333 (5)	−0.0016 (4)	−0.0080 (4)	−0.0020 (4)
O4	0.0123 (4)	0.0210 (5)	0.0223 (4)	−0.0010 (3)	−0.0038 (4)	0.0031 (4)
O5	0.0175 (5)	0.0214 (5)	0.0221 (4)	−0.0027 (4)	0.0013 (4)	0.0028 (4)

Geometric parameters (Å, °)

C1—C2	1.380 (2)	C10—O4	1.4834 (16)
C1—C6	1.3942 (19)	C10—C13	1.517 (2)
C1—H1A	0.9300	C10—C12	1.520 (2)
C2—C3	1.3997 (19)	C10—C11	1.524 (2)
C2—H2A	0.9300	C11—H11A	0.9600
C3—C4	1.3915 (19)	C11—H11B	0.9600
C3—C7	1.5079 (18)	C11—H11C	0.9600
C4—C5	1.391 (2)	C12—H12A	0.9600
C4—H4A	0.9300	C12—H12B	0.9600
C5—C6	1.3893 (19)	C12—H12C	0.9600
C5—H5A	0.9300	C13—H13A	0.9600
C6—O1	1.3653 (16)	C13—H13B	0.9600
C7—C8	1.5408 (19)	C13—H13C	0.9600
C7—H7A	0.9700	C14—O2	1.4578 (17)
C7—H7B	0.9700	C14—H14A	0.9600
C8—N1	1.4415 (16)	C14—H14B	0.9600
C8—C15	1.5265 (18)	C14—H14C	0.9600
C8—H8A	0.9800	C15—O3	1.2079 (18)
C9—O5	1.2210 (17)	C15—O2	1.3309 (17)
C9—O4	1.3424 (16)	N1—H1B	0.8600
C9—N1	1.3447 (17)	O1—H1C	0.8200
C2—C1—C6	119.69 (13)	O4—C10—C11	111.29 (12)
C2—C1—H1A	120.2	C13—C10—C11	110.79 (12)
C6—C1—H1A	120.2	C12—C10—C11	112.37 (13)
C1—C2—C3	121.55 (13)	C10—C11—H11A	109.5
C1—C2—H2A	119.2	C10—C11—H11B	109.5
C3—C2—H2A	119.2	H11A—C11—H11B	109.5
C4—C3—C2	117.90 (12)	C10—C11—H11C	109.5
C4—C3—C7	121.49 (12)	H11A—C11—H11C	109.5
C2—C3—C7	120.51 (12)	H11B—C11—H11C	109.5
C5—C4—C3	121.20 (12)	C10—C12—H12A	109.5
C5—C4—H4A	119.4	C10—C12—H12B	109.5
C3—C4—H4A	119.4	H12A—C12—H12B	109.5
C6—C5—C4	119.85 (12)	C10—C12—H12C	109.5

C6—C5—H5A	120.1	H12A—C12—H12C	109.5
C4—C5—H5A	120.1	H12B—C12—H12C	109.5
O1—C6—C5	122.90 (12)	C10—C13—H13A	109.5
O1—C6—C1	117.34 (12)	C10—C13—H13B	109.5
C5—C6—C1	119.75 (12)	H13A—C13—H13B	109.5
C3—C7—C8	114.62 (10)	C10—C13—H13C	109.5
C3—C7—H7A	108.6	H13A—C13—H13C	109.5
C8—C7—H7A	108.6	H13B—C13—H13C	109.5
C3—C7—H7B	108.6	O2—C14—H14A	109.5
C8—C7—H7B	108.6	O2—C14—H14B	109.5
H7A—C7—H7B	107.6	H14A—C14—H14B	109.5
N1—C8—C15	114.93 (11)	O2—C14—H14C	109.5
N1—C8—C7	109.87 (11)	H14A—C14—H14C	109.5
C15—C8—C7	107.10 (10)	H14B—C14—H14C	109.5
N1—C8—H8A	108.2	O3—C15—O2	123.75 (13)
C15—C8—H8A	108.2	O3—C15—C8	121.54 (12)
C7—C8—H8A	108.2	O2—C15—C8	114.67 (11)
O5—C9—O4	125.14 (13)	C9—N1—C8	121.70 (11)
O5—C9—N1	124.15 (13)	C9—N1—H1B	119.2
O4—C9—N1	110.71 (12)	C8—N1—H1B	119.2
O4—C10—C13	101.82 (11)	C6—O1—H1C	109.5
O4—C10—C12	109.26 (11)	C15—O2—C14	114.55 (12)
C13—C10—C12	110.83 (13)	C9—O4—C10	121.33 (11)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1B \cdots O3 ⁱ	0.86	2.27	3.0583 (16)	153
O1—H1C \cdots O5 ⁱⁱ	0.82	1.92	2.7356 (15)	180

Symmetry codes: (i) $x+1/2, -y+3/2, -z$; (ii) $x, y+1, z$.